NASA-CR-72177

ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

Quarterly Progress Report No. 6
For Quarter Ending October 15, 1966

By

R. W. HARRISON
and
E. E. HOFFMAN

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT NAS 3-6474

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC
CINCINNATI, OHIO 45215

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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

QUARTERLY PROGRESS REPORT 6

Covering the Period
July 15, 1966 to October 15, 1966

Edited by
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Project Metallurgist

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Prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Lewis Research Center

Under Contract NAS 3-6474

October 24, 1966

Technical Management
NASA - Lewis Research Center
Space Power Systems Division
R. L. Davies

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

FOREWORD

The work described herein is sponsored by the National Aeronautics and Space Administration under Contract NAS 3-6474. For this program, Mr. R. L. Davies is the NASA Project Manager.

The program is being administered for the General Electric Company by Dr. J. W. Semmel, Jr., and E. E. Hoffman, is acting as the Program Manager. J. Holowach, the Project Engineer, is responsible for the loop design, facilities, procurement and test operations. R. W. Harrison, the Project Metallurgist, is responsible for the materials procurement, utilization and evaluation aspects of the program. Personnel making major contributions to the program during the current reporting period include:

Alkali Metal Purification and Handling - Dr. R. B. Hand, L. E. Dotson, H. Bradley and J. R. Reeves

Welding and Joining - W. R. Young

Refractory Alloy Procurement - R. G. Frank and L. B. Engel

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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

I. INTRODUCTION

This report covers the period from July 15, 1966 to October 15, 1966, of a program to fabricate, operate for 10,000 hours, and evaluate a potassium corrosion test loop constructed of T-111 (Ta-8W-2Hf) alloy. Materials for evaluation in the turbine simulator include Mo-TZC and Cb-132M. The loop design will be similar to the Prototype Loop; a two-phase, forced convection, potassium corrosion test loop which has been developed under Contract NAS 3-2547. Lithium will be heated by direct resistance in a primary loop. Heat rejection for condensation in the secondary loop will be accomplished by radiation in a high vacuum environment to the water cooled chamber. The compatibility of the selected materials will be evaluated at conditions representative of space electric power system operating conditions, namely:

a. Boiling temperature, 2050°F

b. Superheat temperature, 2150°F

c. Condensing temperature, 1400°F

d. Subcooling temperature, 1000°F

e. Mass flow rate, 40 lb/hr

f. Boiler exit vapor velocity, 50 ft/sec

g. Average heat flux in plug (0-18 inches), 240,000 BTU/hr ft^2

h. Average heat flux in boiler (0-250 inches), 23,000 BTU/hr ft^2

II. SUMMARY

During the sixth quarter of the program, work proceeded on the topics abstracted below:

Significant effort was made in the past quarter in monitoring the fabrication of the advanced refractory alloys. Production of most of the T-111 alloy tubing was completed and delivery is expected by November 1, 1966. All the Mo-TZC and Cb-132M materials have been received.

Assembly of the lithium still is in progress.

A study has been initiated to evaluate the corrosion resistance of advanced tantalum alloys, ASTAR 811 and ASTAR 811CN, to potassium and lithium.

III. PROGRAM STATUS

A. MATERIALS PROCUREMENT

1. T-111 Alloy

The processing of T-111 alloy tubing for Corrosion Loop I (T-111) continued at the Wolverine Tube Division of Calumet-Hecla. As described in Quarterly Progress Report No. 5[1], six base tubes were successfully given a 50 percent reduction to an intermediate tube size. During this report interim the remaining two base tubes were successfully given a similar reduction. Following this first tube reduction, a brief study was performed at General Electric to aid in selecting the annealing temperature to be used on the reduced tube prior to further tube reducing. Samples from the reduced tubes were annealed for one hour at 2400°, 2500°, 2600°, 2700°, 2800°, 2900° and 3000°F in vacuum. The samples were evaluated by means of metallographic examination and microhardness techniques.

Recovery and/or recrystallization have occurred in the annealed specimens shown in Figures 1 through 4. The microhardness and grain size data are presented in Table I. A one-hour anneal at 2700°F was selected to obtain a desirable combination of hardness and grain size. Subsequently, the eight reduced tubes were cleaned, wrapped in tantalum foil, and annealed for one hour at 2700°F in a pressure of less than 1 x 10^{-5} torr at Wolverine Tube.

After this first reduction and anneal, three of four tubes were given a second reduction of approximately 50% to produce the 1.00-inch OD x 0.100-inch wall tubing. A fourth tube requires this additional reduction.

Four other tubes were reduced two more times with approximately a 50% reduction each time, to produce the 0.375-inch OD x 0.065-inch wall tubing. Between these two reductions, the 0.375-inch OD tubing was vacuum annealed in the same manner as described earlier. After cleaning and wrapping in tantalum foil, both the 1.00-inch OD and 0.375-inch OD tubing were given a final anneal

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(1) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 5 for Period Ending July 15, 1966, NASA Contract NAS 3-6474, NASA-CR-72057, pp 3.

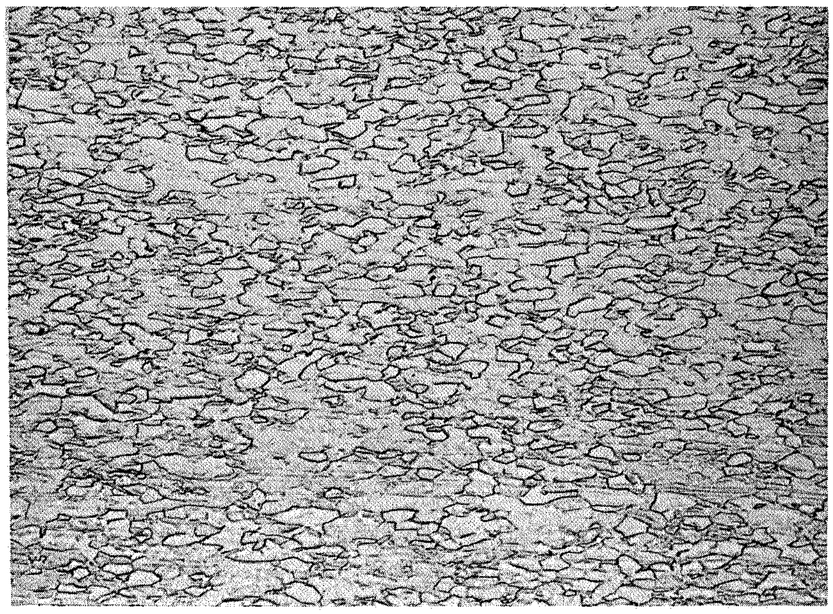
TABLE I. MICROHARDNESS AND GRAIN SIZE OF UNANNEALED AND ANNEALED[a] T-111 ALLOY HEAT NUMBER 111-D-1670 AFTER FIRST REDUCTION

Annealing Temperature °F	Microhardness[b] DPH	ASTM Grain Size[c]
As Reduced	276	8.0
2400	241	8.0
2500	227	7.5
2600	212	7.0
2700	204	7.0
2800	207	6.5
2900	202	6.0
3000	203	6.0

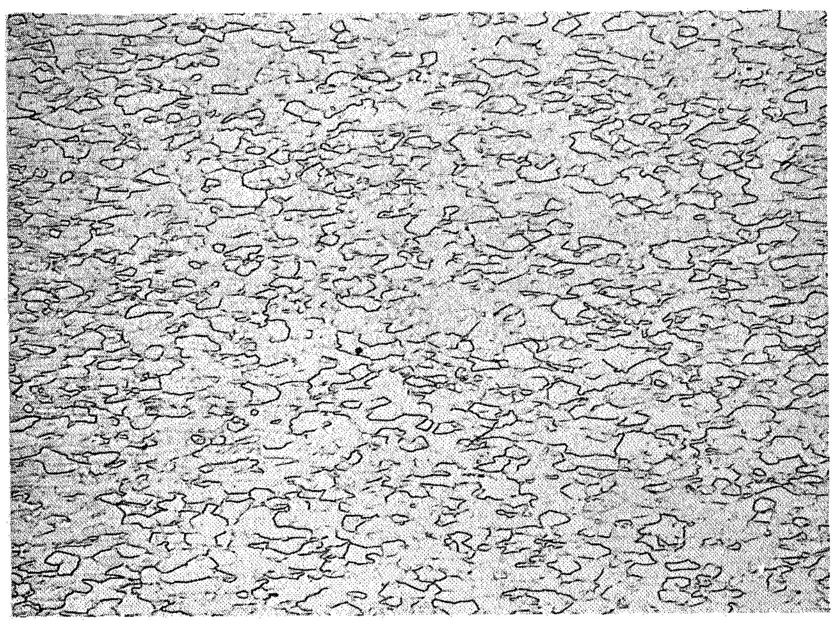
(a) Specimens annealed for one hour at pressures less than 1×10^{-5} torr.

(b) Average of four impressions at mid-wall location: 500-gram load.

(c) Intercept (or Heyn) procedure used.



a) As Reduced



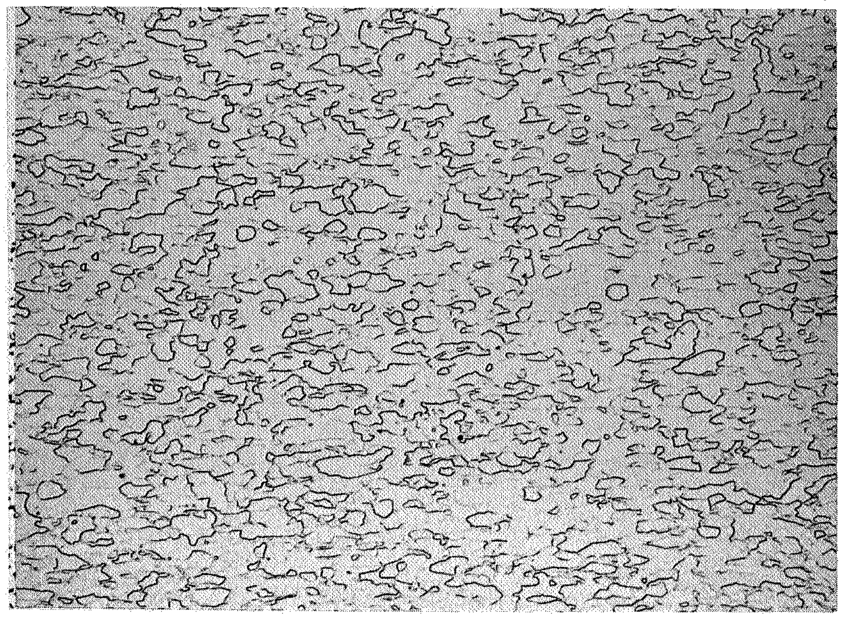
C1127-1

b) Annealed, 2400°F — 1 Hour

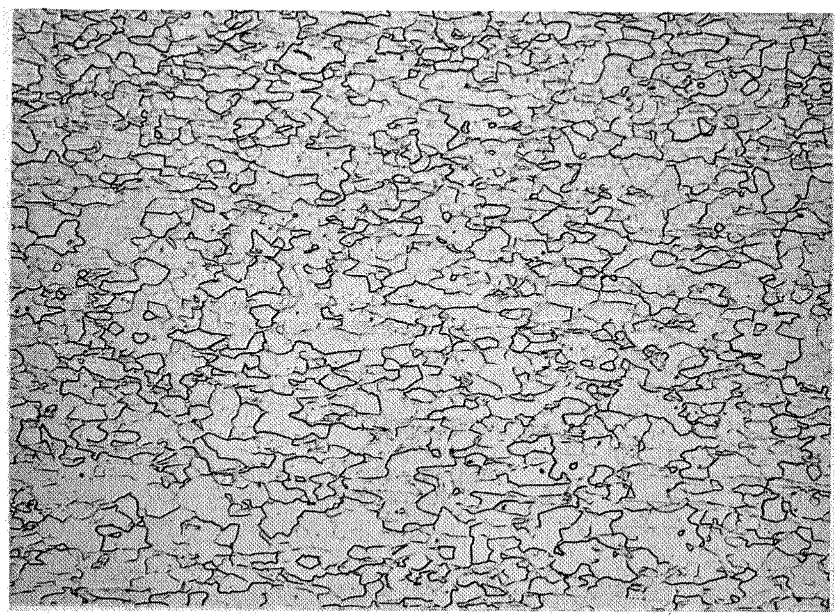
Figure 1. Longitudinal Microstructure of T-111 Alloy Tubing After First Reduction.

Etchant: 30gmNH_4F-20mlH_2O-50mlHNO_3 Mag.: 100X

a)C100121 b)C100221



a) Annealed, 2500°F – 1 Hour



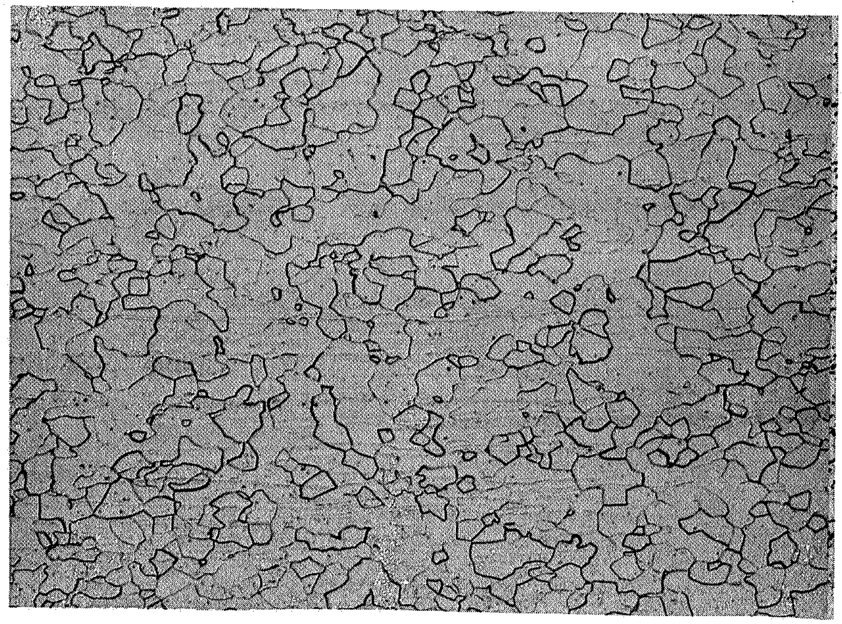
C1127-2

b) Annealed, 2600°F – 1 Hour

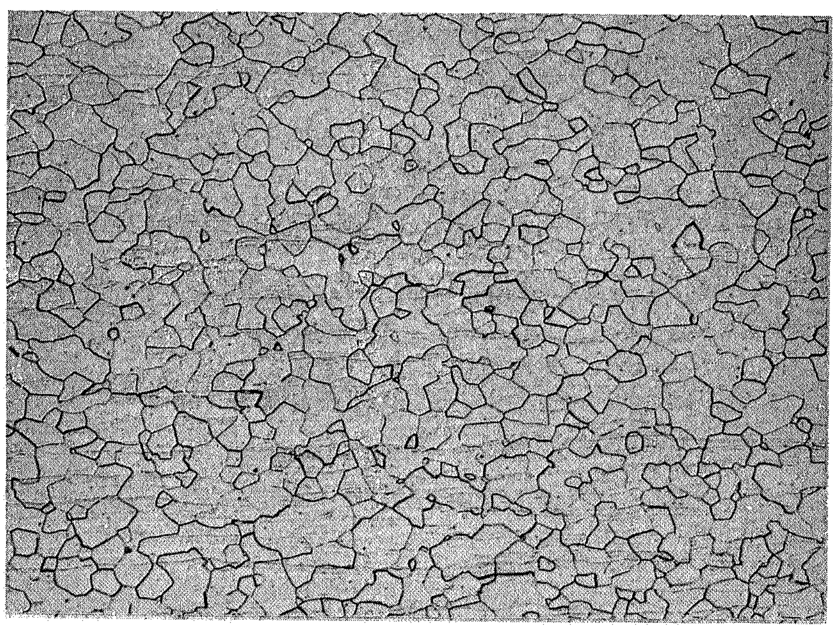
Figure 2. Longitudinal Microstructure of T-111 Alloy Tubing After First Reduction.

Etchant: 30gmNH_4F-20mlH_2O-50mlHNO_3 Mag.: 100X

a)C100321 b)C100421



a) Annealed, 2700°F — 1 Hour



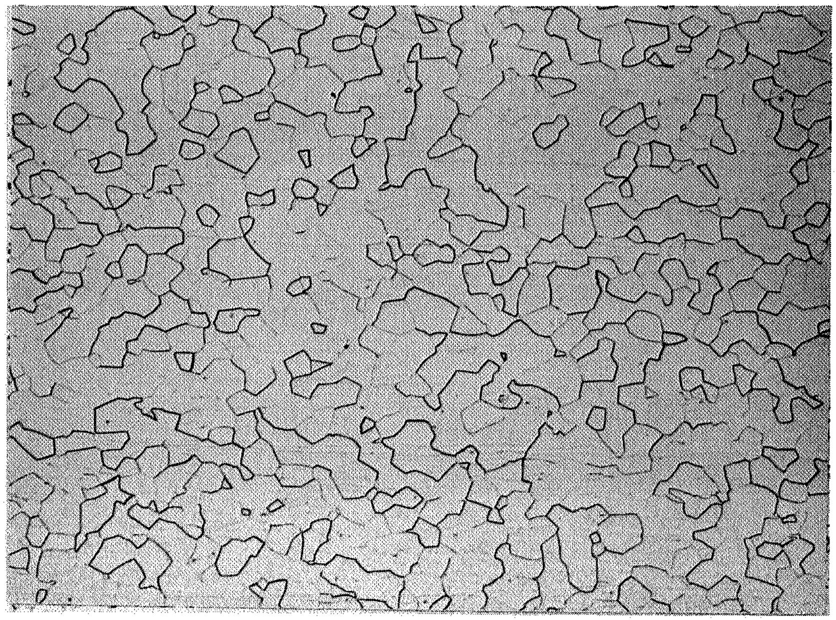
C1127-3

b) Annealed, 2800°F — 1 Hour

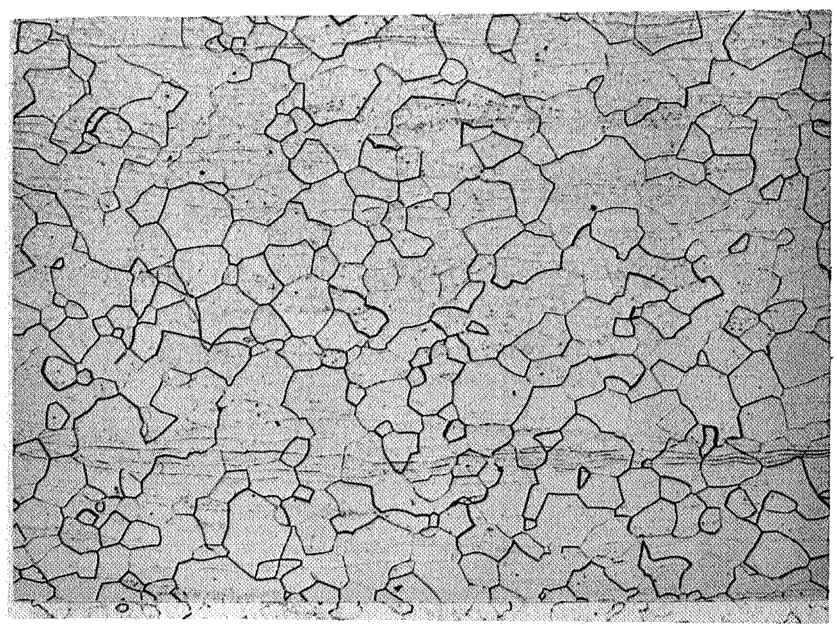
Figure 3. Longitudinal Microstructure of T-111 Alloy Tubing After First Reduction.

Etchant: $30gmNH_4F-20mlH_2O-50mlHNO_3$ Mag.: 100X

a)C100521 b)C100621



a) Annealed, 2900°F — 1 Hour



C1127-4

b) Annealed, 3000°F — 1 Hour

Figure 4. Longitudinal Microstructure of T-111 Alloy Tubing After First Reduction.

Etchant: $30gmNH_4F$-$20mlH_2O$-$50mlHNO_3$ Mag.: 100X

a)C100721 b)C100821

for one hour at 3000°F at a pressure of less than 1 x 10^{-5} torr at Wolverine Tube. Final inspection of the tubing is now in progress and delivery of the tubing is expected by November 1, 1966.

The products made from the fourth and fifth T-111 alloy ingots (No. 111-D-1102 and 111-D-1765, respectively) have been completed and are awaiting final vacuum heat treatment at 3000°F for one hour at The Boeing Company, Seattle, Washington.

Electron beam melting of a sixth ingot, which was necessary when a poor yield was obtained from the fifth ingot, was completed at Fansteel on July 18, 1966. The sixth EB melted Ta-8W alloy ingot was rolled into 4.0-inch diameter electrode stock on August 11, 1966, at Braeburn Alloy Steel. Vacuum arc melting of the T-111 alloy composition was completed on September 14, 1966, at Fansteel. This ingot is scheduled for extrusion at Canton Drop Forging Company on October 21, 1966. The material to be obtained from this ingot is for back-up purposes and is not critical to initiating the loop fabrication phase of the program.

2. Cb-132M Alloy

The 2.25-inch diameter Cb-132M alloy billet was machined into 2.0-inch diameter rod.

The 1.72-inch billet was decanned, inspected, and machined into 1.0-inch diameter rod.

The testing and inspection of the Cb-132M alloy material has been completed and the material has been received. The chemical analyses of the ingot and stress-relieved rod, as supplied by Universal Cyclops, are shown in Table II. The mechanical properties, also supplied by Universal Cyclops, are presented in Table III. A study is being performed by General Electric on the 2-inch diameter Cb-132M alloy rod to determine whether a thermal treatment will significantly improve the room temperature ductility without appreciable recrystallization or reduction in the yield strength.

3. Mo-TZC Alloy

A study is also being performed on the Mo-TZC alloy to define an annealing treatment which will improve the room temperature ductility without appreciable recrystallization or reduction in the yield strength.

TABLE II. CHEMICAL ANALYSES OF Cb-132M ALLOY (HEAT NO. 66-95119) PRODUCED BY UNIVERSAL CYCLOPS STEEL CORPORATION

	Material					
	Ingot				2-Inch Dia. Rod	
	Top-Center	Top-Mid-radius	Top-Edge	Bottom-Center	Center	Edge
%						
C	0.13	0.14	0.15	0.13	0.124	0.119
Ta	19.82	19.53	19.44	20.36	--	--
W	14.9	13.9	14.0	15.3	--	--
Mo	5.01	5.06	5.02	4.72	--	--
Zr	1.97	2.13	2.16	1.75	--	--
ppm						
O	70	<50	<50	<50	28	35
N	30	30	35	40	76	82
H	2.8	2.1	1.9	2.5	4.0	3.4
Al	<20	<20	<20	<20	--	--
B	<1	<1	<1	<1	--	--
Cd	<5	<5	<5	<5	--	--
Co	<10	<10	<10	<10	--	--
Cr	<20	<20	<20	<20	--	--
Cu	<40	<40	<40	<40	--	--
Fe	<50	50	50	50	--	--
Mg	<20	<20	<20	<20	--	--
Mn	<20	<20	<20	<20	--	--
Ni	<20	<20	<20	<20	--	--
Pb	<20	20	<20	<20	--	--
Si	<50	<50	<50	<50	--	--
Sn	30	100	25	35	--	--
Ti	<40	<40	<40	<40	--	--
V	<20	<20	<20	<20	--	--

TABLE III. MECHANICAL PROPERTIES[a] OF Cb-132M ALLOY (HEAT NO. 66-95119) PRODUCED BY UNIVERSAL CYCLOPS STEEL CORPORATION

Specimen Location	Room Temperature Tensile Properties[b]: Ultimate Strength 1,000 psi	0.2% Yield Strength 1,000 psi	Reduction in Area %	Elong. %	Stress-Rupture Life at 2200°F and 30,000 psi in Vacuum: Life Hours	Reduction in Area %	Elong. %	Vacuum at Test Temp. Torr
1-Inch Diameter Rod[c]	139.0	115.0	14	11	10.4	43	32	2.9×10^{-6}
	138.5	116.5	13	11	10.1	42	19	1.7×10^{-6}
2-Inch Diameter Rod	130.0[c,d]	116.5	3.0	2	78.4[e]	40	25	7×10^{-7}
	132.0[c,d]	116.4	1.5	2	38.1[f,c]	42	15	9×10^{-6}

(a) Tensile and stress-rupture specimens were machined from Cb-132M alloy rod following annealing for one hour at 2400°F The axes of the specimens were parallel to the rolling direction.

(b) Tensile properties were determined using a strain rate of 0.005 inch/inch/min up to 0.2% offset and then 0.050 inch/min head rate to fracture.

(c) The specimens had a 0.160-inch diameter with a 0.80-inch gauge length.

(d) Specimen failed in the radius.

(e) Total hours, specimen adapter failed after 37.9 hours and test was restarted after replacement of adapter. The specimen had a 0.250-inch diameter with a 1.45-inch gauge length.

(f) Total hours, threads failed on specimen after 11.6 hours and test was restarted after modifying specimen to buttonhead design.

B. ALKALI METAL PURIFICATION AND HANDLING

All the parts for the lithium still were machined and assembly is in progress. The cooling coil has been brazed to the stainless steel condenser tube jacket of the lithium still. The Cb-1Zr tubing for the receiver level probe well was received. Assembly of the lithium still is about 50 percent completed.

C. ADVANCED TANTALUM ALLOY CAPSULE TESTS

A study has been initiated to evaluate the corrosion resistance of two advanced tantalum alloys to potassium and lithium. These alloys, designated ASTAR 811 (Ta-8W-1Hf-1Re) and ASTAR 811CN (Ta-8W-1Hf-1Re-0.012C-0.012N), have been selected by the NASA Program Manager from the compositions currently being developed under NASA contract*. Both alloys will be tested for resistance to corrosion by lithium and potassium in the as-received and oxygen-contaminated (300-500 ppm oxygen) conditions. The stability of the carbon and nitrogen in the ASTAR 811CN alloy in a lithium environment also will be evaluated. Each alloy will be exposed in four conditions to potassium liquid and vapor in a weld segment reflux capsule shown schematically in Figure 5. The wall segments of this capsule are comprised of each alloy in each of four conditions namely: welded sheet, welded and postweld annealed sheet, contaminated and welded sheet, and contaminated, welded and postweld annealed sheet. These capsules will be tested for 2,500 hours at 2100°F.

Bend test specimens of each of the alloys in the forementioned conditions will be exposed to lithium in an individual T-111 alloy thermal convection capsule, shown schematically in Figure 6. This design incorporates a T-111 alloy double wall vacuum insulated inner tube to assist in establishing a thermal gradient which will result in convection flow of the lithium. The maximum lithium temperature will be 2300°F. The alloy specimens are located along the length of the annulus between the capsule wall and this inner tube. These capsules also will be tested for 2,500 hours.

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* Development of Dispersion Strengthened Tantalum Base Alloy, Astronuclear Laboratories, Westinghouse Electric Company, NASA Contract NAS 3-2542.

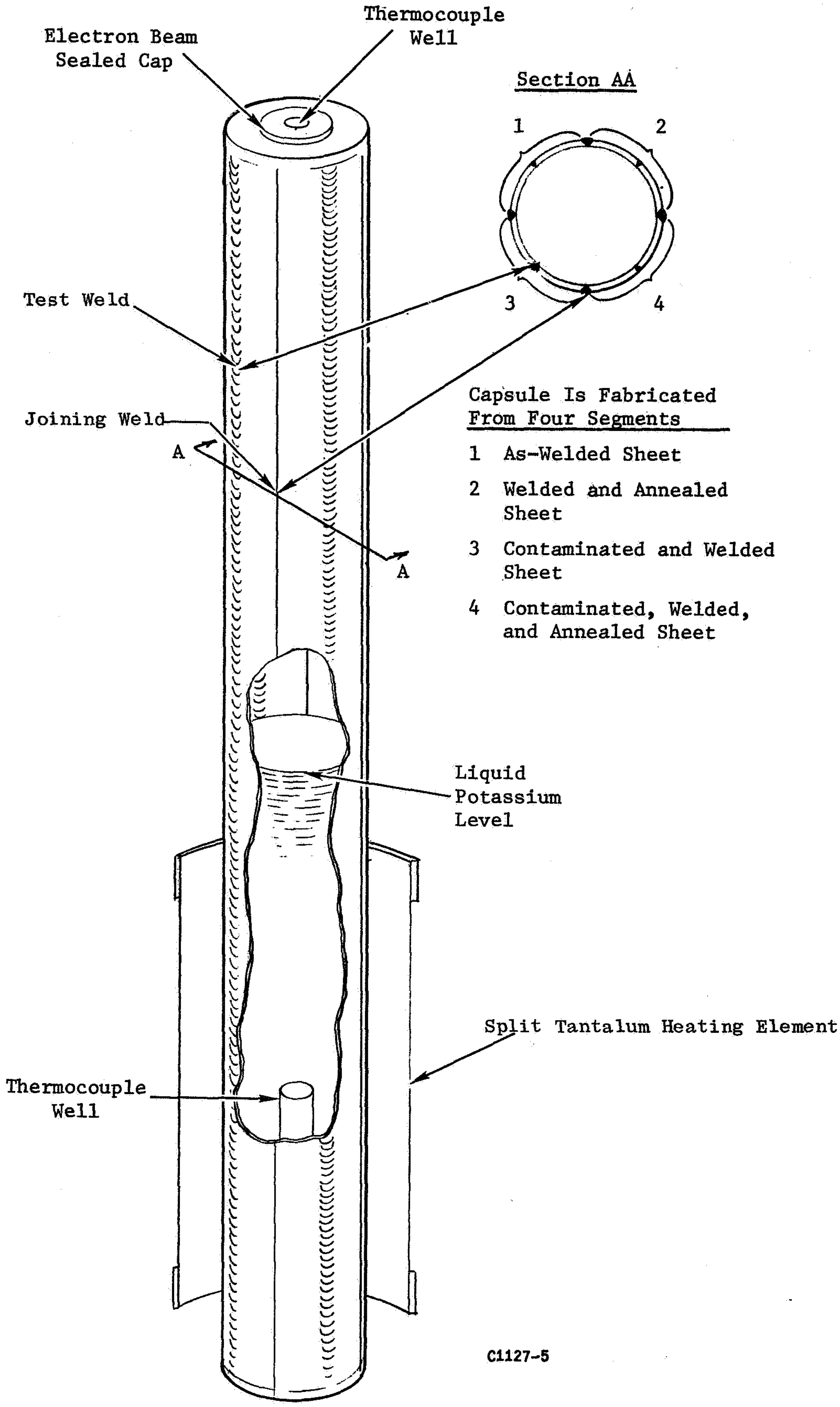

Figure 5. Welded Segment Refluxing Potassium Capsule.

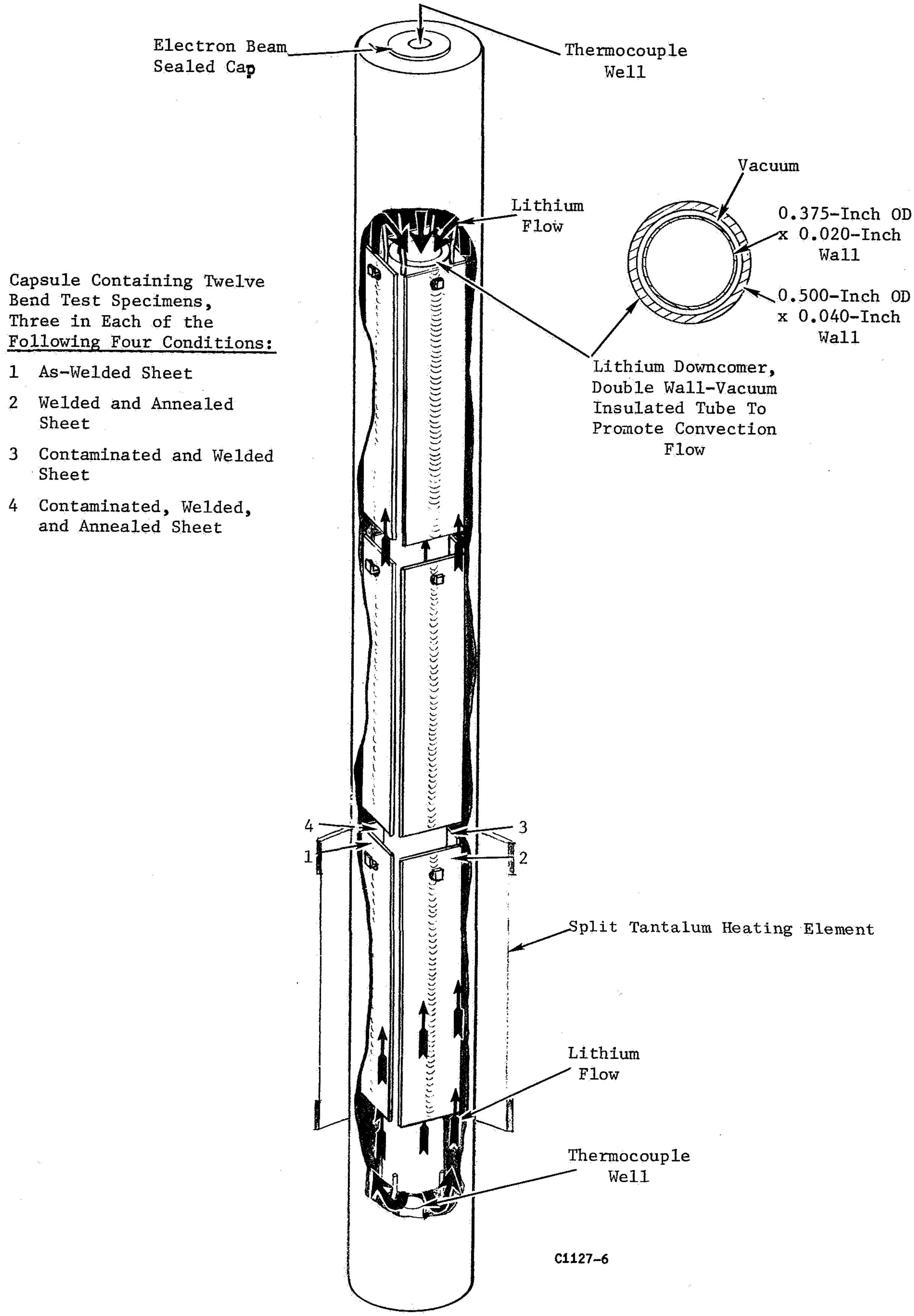

Figure 6. Lithium Thermal Convection Capsule Containing Twelve Bend Specimens.

A similar capsule design shown schematically in Figure 7, will be used to determine the extent of carbon and nitrogen migration between the hottest and coolest ASTAR 811CN alloy specimens in a lithium environment. The capsule and six stress-rupture specimens contained therein will be fabricated from the ASTAR 811CN alloy. Again, a T-111 alloy inner tube will be used to enhance the lithium flow. After 2,500 hours exposure at a maximum temperature of 2300°F, the specimens and capsule will be examined for possible carbon and/or nitrogen mass transfer.

All necessary materials for this study have been ordered, and the capsules and associated test facility designs have been completed.

A brief oxygen contamination study was performed on T-111 alloy specimens to establish time-temperature-pressure criteria for subsequent contamination of the ASTAR 811 and ASTAR 811CN alloys which are to be used in the preparation of specimens for the capsule tests illustrated in Figures 5 and 6. The kinetics of oxygen solutioning in the low ppm range for these alloys should not vary considerably as the compositions of these alloys are similar.

The T-111 alloy contamination exposures were performed in the 24-inch diameter by 54-inch high Varian high vacuum system shown in Figure 8. The 0.040-inch sheet specimens, measuring 12 inches long x 1 inch wide, were heated by direct resistance, and the temperature was monitored with W+25%Re/W+3%Re thermocouples as shown in Figure 9. The chamber was initially evacuated by means of a 260 liter/sec turbomolecular pump. Following bakeout of the system, a cold wall pressure of 1.9×10^{-9} torr was achieved. Three specimens were exposed at 2400°F to a 1×10^{-5} torr pressure, maintained by means of an oxygen leak, for 25, 50 and 100 hours.

Post test chemical analysis indicated the oxygen concentrations of these specimens to be 5,100 ppm, 6,940 ppm, and 8,420 ppm, respectively, as compared to a pretest analysis of 89 ppm. This indicated that exposures at 1×10^{-6} torr and 2400°F would lead to oxygen concentrations of 300 to 500 ppm at convenient exposure times. A fourth T-111 alloy specimen was exposed for 30 hours under these conditions, and post test analysis indicated the oxygen concentration to be 442 ppm. The ASTAR 811 and ASTAR 811CN contaminated specimens will be prepared in this facility under the latter time-temperature-pressure conditions.

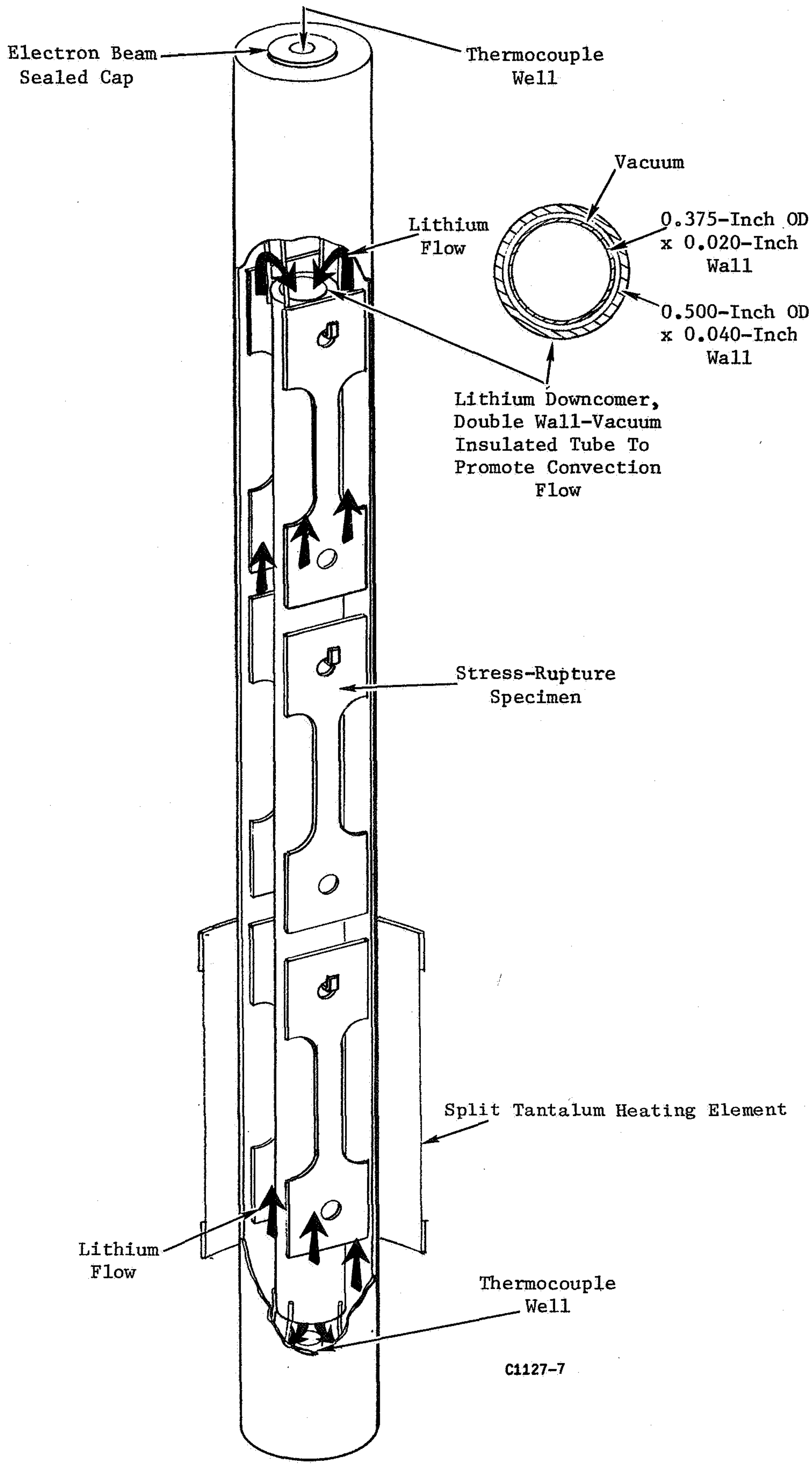

Figure 7. Lithium Thermal Convection Capsule Containing Six Stress-Rupture Specimens.

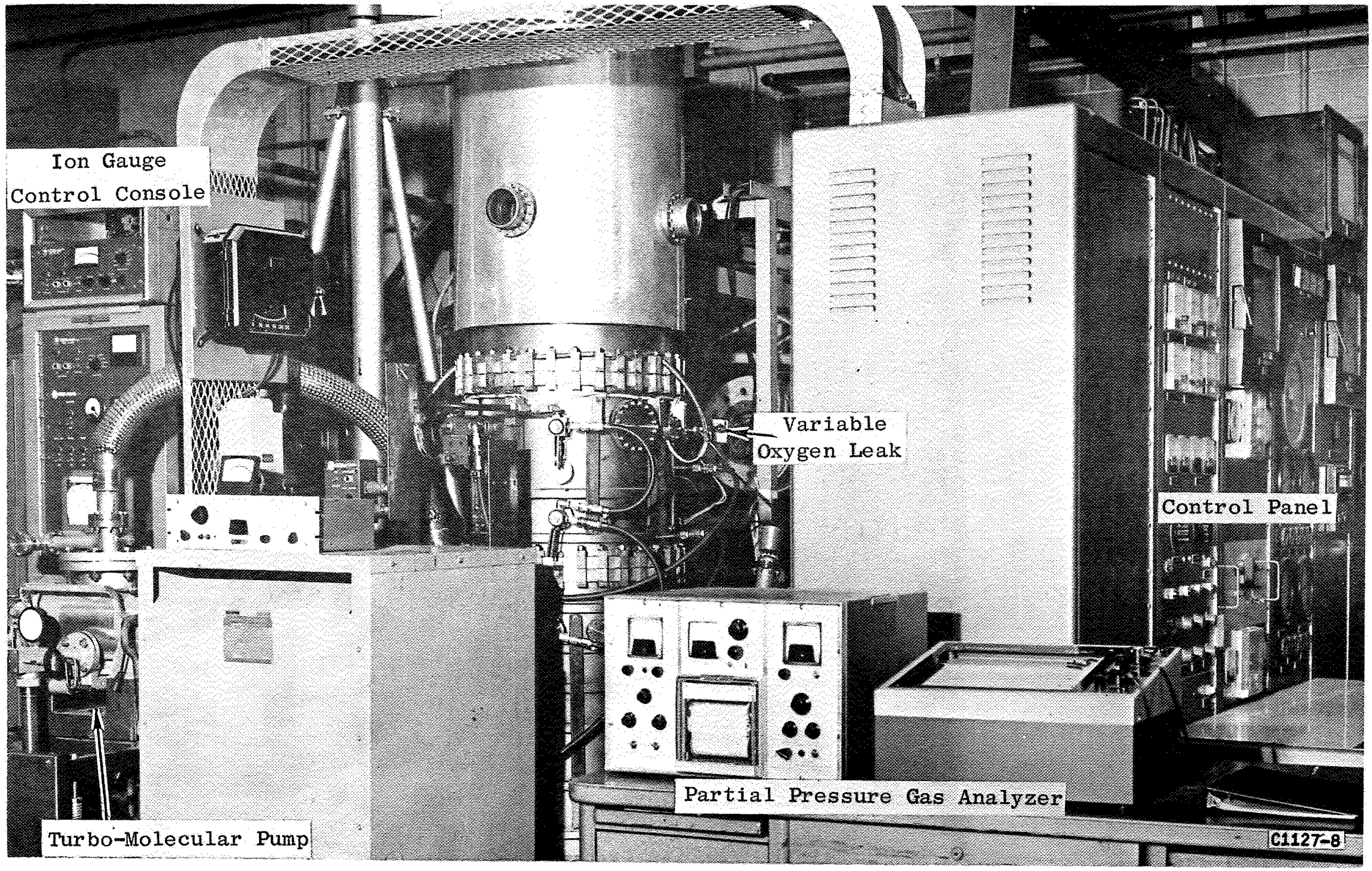

Figure 8. High Vacuum System (10^{-10} Torr Range) Used for the Contamination of T-111 Alloy Specimens. The Chamber is 24 Inches in Diameter and 54 Inches High and Incorporates a 1000 liter/sec. Getter-Ion Pump. The Oxygen Partial Pressure is Controlled by a Variable Leak. (C66080914)

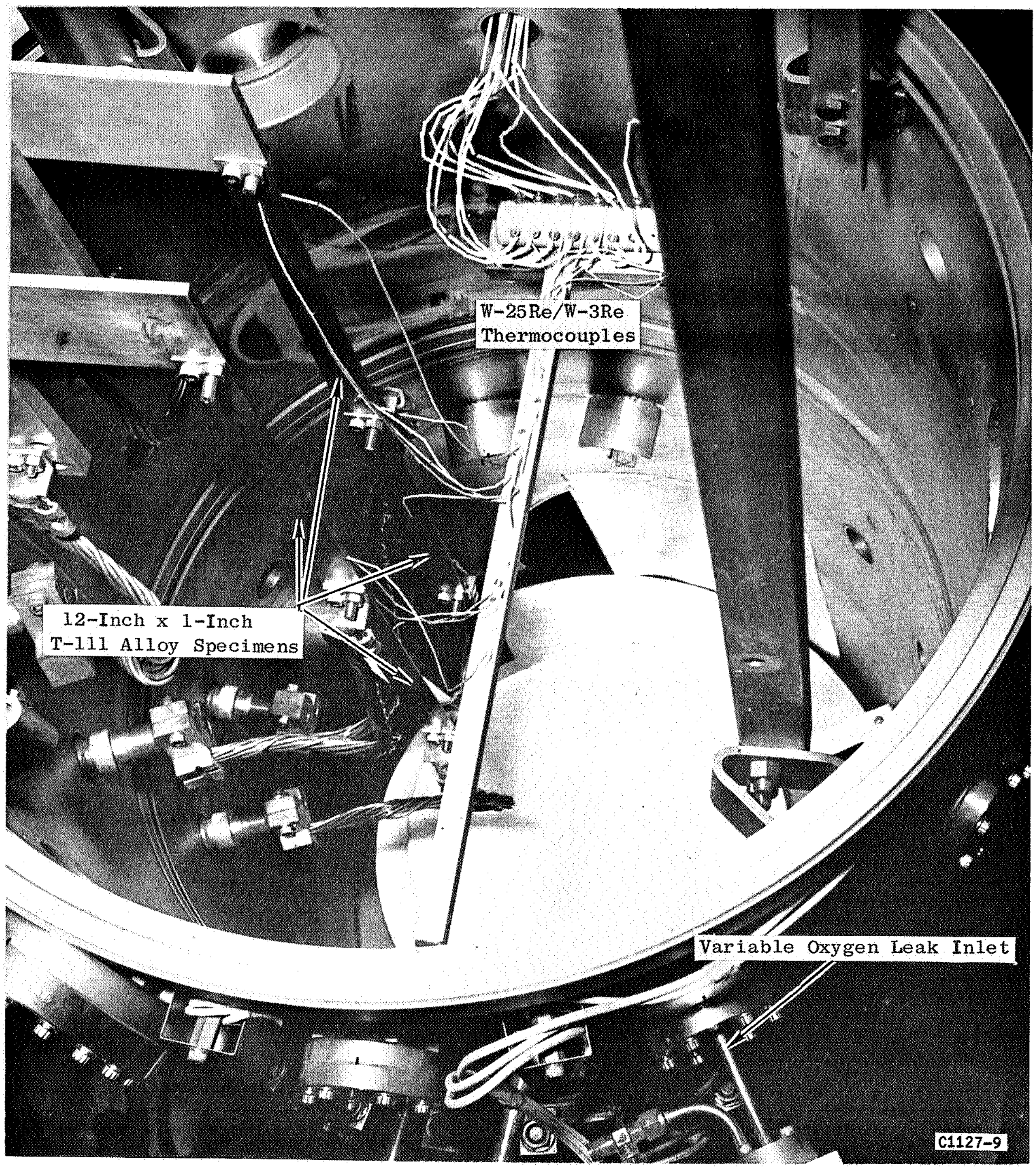

Figure 9. T-111 Alloy Contamination Specimen Installation. (C66080382)

IV. FUTURE PLANS

A. All the refractory alloys materials for Corrosion Loop I (T-111) will be received, inspected, and released for manufacturing loop components.

B. Fabrication of loop components will be initiated.

C. Fabrication and installation of the lithium still into the purification system will be completed and distillation of the lithium will be initiated.

PUBLISHED REPORTS

Quarterly Progress	For Quarter Ending
Report No. 1 (NASA-CR-54477)	July 15, 1965
Report No. 2 (NASA-CR-54845)	October 15, 1965
Report No. 3 (NASA-CR-54911)	January 15, 1966
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